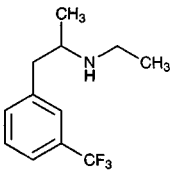
OTHER SUBSTANCES

Also analyzed: acebutolol, acepromazine, acetophenazine, N-acetylprocainamide, albuterol, alprenolol, amethocaine, amiodarone, amitriptyline, antazoline, atenolol, azacyclonal, bamethan, benactyzine, benperidol, benzethidine, benzocaine, benzoctamine, benzphetamine, benzquinamide, bromhexine, bromodiphenhydramine, bromperidol, brompheniramine, brompromazine, buclizine, bufotenine, bupivacaine, buprenorphine, butacaine, butethamate, chlorcyclizine, chlorpheniramine, chlorphenoxamine, chlorprenaline, chlorpromazine, chlorprothixene, cimetidine, cinchonidine, cinnarizine, clemastine, clomipramine, clonidine, cocaine, cyclazocine, cyclizine, cyclopentamine, cyproheptadine, deserpidine, desipramine, dextromoramide, dextropropoxyphene, dicyclomine, diethylcarbamazine, diethylpropion, diethylthiambutene, dihydroergotamine, dimethindene, dimethothiazine, diphenhydramine, diphenoxylate, dipipanone, diprenorphine, dipyridamole, disopyramide, dothiepin, doxapram, doxepin, doxylamine, droperidol, ephedrine, ergocornine, ergocristine, ergocristinine, ergocryptine, ergometrine, ergosine, ergosinine, ergotamine, ethopropazine, etorphine, etoxeridine, fenfluramine, fenoterol, fentanyl, flavoxate, fluopromazine, flupenthixol, fluphenazine, flurazepam, haloperidol, hydroxyzine, hyoscine, ibogaine, imipramine, indapamine, iprindole, isothipendyl, isoxsuprine, ketanserin, laudanosine, lidocaine, lofepramine, loxapine, maprotiline, mecamylamine, meclophenoxate, meclozine, medazepam, mephentermine, mepivacaine, meptazinol, mepyramine, mesoridazine, metaraminol, methadone, methamphetamine, methapyrilene, methdilazene, methotrimeprazine, methoxamine, methoxyphenamine, methoxypromazine, methylephedrine, methylergonovine, methysergide, metoclopramide, metopimazine, metoprolol, mianserin, morazone, nadolol, nalorphine, naloxone, naphazoline, nicotine, nifedipine, nomifensine, nortriptyline, noscapine, orphenadrine, oxeladin, oxprenolol, oxymetazolin, papaverine, pargyline, pecazine, penbutolol, pentazocine, penthienate, pericyazine, perphenazine, phenadoxone, phenampromide, phenazocine, phenbutrazate, phendimetrazine, phenelzine, phenglutarimide, phenindamine, pheniramine, phenmetrazine, phenomorphan, phenoperidine, phenothiazine, phenoxybenzamine, phentolamine, phenylephrine, phenyltoloxamine, physostigmine, piminodine, pimozide, pindolol, pipamazine, pipazethate, piperacetazine, piperidolate, pipradol, pirenzepine, piritramide, pizotifen, practolol, pramoxine, prazosin, prenylamine, prilocaine, primaquine, proadifen, procainamide, procaine, prochlorperazine, procyclidine, proheptazine, prolintane, promazine, promethazine, pronethalol, properidine, propiomazine, propranolol, prothipendyl, protriptyline, proxymetacaine, pseudoephedrine, pyrimethamine, quinidine, quinine, ranitidine, rescinnamine, sotalol, tacrine, terazosin, terbutaline, terfenadine, thenyldiamine, theophylline, thiethylperazine, thiopropazate, thioproperazine, thioridazine, thiothixene, thonzylamine, timolol, tocainide, tolpropamine, tolycaine, tranylcypromine, trazodone, trifluoperazine, trifluperidol, trimeperidine, trimeprazine, trimethobenzamide, trimethoprim, trimipramine, tripelennamine, triprolidine, tryptamine, verapamil, xylometazoline

REFERENCE

Jane,I.; McKinnon,A.; Flanagan,R.J. High-performance liquid chromatographic analysis of basic drugs on silica columns using non-aqueous ionic eluents. II. Application of UV, fluorescence and electrochemical oxidation detection, *J.Chromatogr.*, **1985**, *323*, 191–225.

Fenfluramine



Molecular formula: $C_{12}H_{16}F_3N$
Molecular weight: 231.26
CAS Registry No.: 458-24-2, 404-82-0 (HCl)
Merck Index: 4015
Lednicer No.: 1 70

SAMPLE

Matrix: blood

Sample preparation: 1 mL Plasma + 1 mL 0.6 M pH 9.8 carbonate buffer + 40 µL 5 µg/mL maprotiline in 10 mM HCl + 5 mL 200 g/L ethyl acetate in n-heptane, mix by rocking for 10 min, centrifuge at 1500 g for 10 min. Remove organic layer and add it to 150 µL 100 mM HCl, mix 10 min, centrifuge at 1500 g for 10 min. Discard organic layer and evaporate aqueous layer at 45° in a vacuum centrifuge for 1 h. Take up residue in 50 µL 1 M pH 10.3 carbonate buffer and 25 µL 10 mg/mL dansyl chloride in MeCN, vortex, allow to react at room temper-

ature for 45 min, evaporate at 45° in a vacuum centrifuge for 20 min, reconstitute in 125 μL MeCN:water 75:25, vortex, centrifuge for 3-5 min, inject a 25-40 μL aliquot.

HPLC VARIABLES

Column: 250 × 4.6 5 μm Supelcosil LC-18
Mobile phase: MeCN:25 mM KH_2PO_4 75:25 + 500 μL/L orthophosphoric acid + 600 μL/L n-butylamine
Flow rate: 2
Injection volume: 25-40
Detector: F ex 235 em 470 (cut-off)

CHROMATOGRAM

Retention time: 8.21
Internal standard: maprotiline (12.8)

OTHER SUBSTANCES

Simultaneous: fluoxetine, propranolol, clovoxamine, fluvoxamine, amoxapine, desipramine, protriptyline, nortriptyline, sertraline, norfluoxetine
Noninterfering: amitriptyline, imipramine, clomipramine, trimipramine, mianserin, chlordiazepoxide, trazodone, cyclobenzaprine, nomifensine, bupropion, metoprolol, atenolol, pindolol, tranylcypromine, moclobemide, thioridazine, citalopram, clozapine, carbamazepine, doxepin, loxapine

KEY WORDS

plasma

REFERENCE

Suckow,R.F.; Zhang,M.F.; Cooper,T.B. Sensitive and selective liquid-chromatographic assay of fluoxetine and norfluoxetine in plasma with fluorescence detection after precolumn derivatization, *Clin.Chem.*, **1992**, *38*, 1756–1761.

SAMPLE

Matrix: blood
Sample preparation: 1 mL Plasma + 100 μL 1.25 μg/mL IS in water + 200 μL water, vortex, add 400 μL 1 M trichloroacetic acid, vortex, centrifuge at 2500 g for 10 min. Remove 900 μL of the supernatant and add it to 250 μL 1 M NaOH, add 2 mL reagent, vortex for 2 s, let stand for 90 min. Remove a 1 mL aliquot of the lower organic layer and evaporate it to dryness under a stream of nitrogen, reconstitute the residue in 200 μL dichloromethane, inject a 35 μL aliquot. (Reagent was 10 μM 3,5-dinitrophenylisocyanate in dichloromethane. Prepare as follows. Stir 6.40 g 3,5-dinitrobenzoyl chloride in 100 mL glacial acetic acid, add 1.80 g sodium azide in small increments, stir for 1 h, add 300 mL cold water. Filter off the 3,5-dinitrobenzyl azide precipitate and wash it with a small portion of water. Dry overnight in a vacuum desiccator. Reflux 25 mg 3,5-dinitrobenzyl azide dissolved in 5 mL toluene for 10 min, cool to room temperature, make up to 50 mL with dichloromethane, dilute an aliquot 1:200 with dichloromethane to give a 10 μM solution of 3,5-dinitrophenylisocyanate. Prepare fresh daily. CAUTION! 3,5-Dinitrobenzyl azide may be explosive and 3,5-dinitrophenylisocyanate may be toxic!)

HPLC VARIABLES

Column: 250 × 4.6 5 μm (R)-naphthylurea Chiral (Supelco)
Mobile phase: Hexane:isopropanol:MeCN 89:9:2
Flow rate: 1.2 for 15 min, 3.5 for 13 min, 1.2 for 7 min
Injection volume: 35
Detector: UV 235

CHROMATOGRAM

Retention time: 10 (d), 11.5 (l)
Internal standard: β-methylphenethylamine (20.7, first peak)
Limit of quantitation: 10 ng/mL

OTHER SUBSTANCES

Extracted: metabolites

KEY WORDS
plasma; conduct analyses under yellow light; chiral; pharmacokinetics; derivatization

REFERENCE
Zeng,J.-N.; Dou,L.; Duda,M.; Stuting,H.H. New chiral high-performance liquid chromatographic methodology used for the pharmacokinetic evaluation of dexfenfluramine, *J.Chromatogr.B*, **1994**, *654*, 231–248.

SAMPLE
Matrix: blood
Sample preparation: 2 mL Whole blood or plasma + 2 mL buffer + 5 mL chloroform:isopropanol: n-heptane 60:14:26, shake gently horizontally for 10 min, centrifuge at 2800 g for 10 min. Remove the lower organic layer and evaporate it to dryness under vacuum at 45°, reconstitute the residue in 100 μL mobile phase, centrifuge at 2800 g for 5 min, inject a 50 μL aliquot of the supernatant. (Buffer was saturated ammonium chloride solution 25% diluted with water, adjusted to pH 9.5 with 25% ammonia solution.)

HPLC VARIABLES
Column: 300 × 3.9 4 μm NovaPack C18
Mobile phase: MeOH:THF:buffer 65:5:30 (Buffer was 0.68 g/L (10 mM (sic)) KH_2PO_4 adjusted to pH 2.6 with concentrated orthophosphoric acid.) (At the end of each session wash the column with water for 1 h and MeOH for 1 h, re-equilibrate for 30 min.)
Column temperature: 30
Flow rate: 0.8
Injection volume: 50
Detector: UV 264

CHROMATOGRAM
Retention time: 5.28
Limit of detection: <120 ng/mL

KEY WORDS
whole blood; plasma; interferences may occur–compounds (all of which are extracted) elute in this order tenoxicam; iproniazid; methocarbamol; methotrexate; caffeine; nialamide; colchicine; cytarabine; benzoylecgonine; acetaminophen; diazoxide; dacarbazine; sulfinpyrazole; flumazenil; sulpride; morphine; atenolol; toloxatone; terbutaline; albuterol; phenobarbital; ranitidine; tiapride; phenol; chlormezanone; aspirin; metformin; ritodrine; codeine; sultopride; amisulpride; naltrexone; lisinopril; benzocaine; nizatidine; nalorphine; mephenesin; naloxone; sotalol; carteolol; procainamide; carbamazepine; bromazepam; nalbuphine; nadolol; procarbazine; dihydralazine; omeprazole; strychnine; acebutolol; glutethimide; chlorpropamide; glipizide; triazolam; prazosin; flunitrazepam; clonazepam; metoclopramide; melphalan; estazolam; tolbutamide; ephedrine; clonidine; pindolol; clobazam; minoxidil; disopyramide; nitrazepam; dextromethorphan; tofisopam; zopiclone; debrisoquine; sulindac; alprazolam; cycloguanil; lorazepam; methaqualone; ketamine; piroxicam; metoprolol; nifedipine; quinine; mephentermine; prilocaine; pentazocine; oxazepam; tiaprofenic acid; quinidine; celiprolol; ajmaline; yohimbine; lidocaine; secobarbital; viloxazine; mepivacaine; meperidine; doxylamine; labetalol; temazepam; amodiaquine; benperidol; droperidol; hydroxychloroquine; zolpidem; ketoprofen; alminoprofen; cicletanine; moclobemide; chloroquine; cocaine; timolol; nomifensine; ticlopidine; acenocoumarol; vindesine; mexiletine; dipyridamole; trazodone; pipamperone; pyrimethamine; benazepril; vincristine; metapramine; chlordiazepoxide; oxprenolol; warfarin; clorazepate; flecainide; phencyclidine; thiopental; fenfluramine; metipranolol; triprolidine; naproxen; buprenorphine; verapamil; buspirone; tianeptine; midazolam; bupivacaine; carbinoxamine; loprazolam; cetirizine; chlorpheniramine; moperone; cibenzoline; medifoxamine; astemizole; vinblastine; nicardipine; bisoprolol; diltiazem; glibornuride; reserpine; aconitine; nitrendipine; diazepam; mianserin; ramipril; haloperidol; tetracaine; alprenolol; acepromethazine; glibenclamide; chlorophenacinone; doxepin; nimodipine; diphenhydramine; cyclizine; histapyrrodine; phenylbutazone; demexiptiline; clozapine; proguanil; trifluperidol; medazepam; cyamemazine; bumadizone; suriclone; propranolol; acepromazine; dothiepin; dextromoramide; fenoprofen; dextropropoxyphene; loxapine; betaxolol; propafenone; promethazine; thioproperazine; methadone; amoxapine; quinupramine; opipramol; cyproheptadine; brompheniramine; mefenidramine; protriptyline; flurbiprofen; tetrazepam; zorubicin; prazepam; alimemazine; loperamide; imipramine; desipramine; levomepromazine; hydroxyzine; niflumic acid; penbutolol; fluvoxamine; pimozide; daunorubicin; indomethacin; maprotiline; tropatenine; etodolac; fluoxetine; amitriptyline; nortriptyline; tioclomarol; diclofenac; mefloquine; trimipramine; chlorambucil;

lidoflazine; ibuprofen; floctafenine; alpidem; loratadine; chlorpromazine; clomipramine; carpipramine; thioridazine; fentiazac; clemastine; mefenamic acid; fluphenazine; prochlorperazine; penfluridol; bepridil; terfenadine; trifluoperazine

REFERENCE

Tracqui,A.; Kintz,P.; Mangin,P. Systematic toxicological analysis using HPLC/DAD, *J.Forensic Sci.*, **1995**, *40*, 254–262.

SAMPLE

Matrix: blood, urine

Sample preparation: Add 1 mL whole blood or urine to Toxi-Tube A (Toxi-Lab, Irvine CA), add 3 mL water, mix by gentle inversion for 5 min, centrifuge at 1500 g for 5 min. Remove the organic layer and evaporate it to dryness under a stream of nitrogen at 40°, reconstitute the residue with 50 μL MeCN:water 50:50, vortex for 10 s, centrifuge at 7500 g for 2 min, inject a 10 (urine) or 30 (blood) μL aliquot. (The detector wavelength shown is the wavelength of maximum absorbance. This will not necessarily be the optimal wavelength for the separation. Multiple wavelengths from 200-350 nm can be scanned using a diode-array detector. Otherwise, 220 nm may be a reasonable choice for initial work. Matrix may interfere.)

HPLC VARIABLES

Guard column: 20 mm long Symmetry C18

Column: 250 × 4.6 5 μm Symmetry C8 (Waters)

Mobile phase: Gradient. A was 50 mM pH 3.8 sodium phosphate buffer. B was MeCN. A:B 85:15 for 6.5 min, 65:35 for 18.5 min, 20:80 for 3 min (step gradient), re-equilibrate at initial conditions for 7 min.

Column temperature: 30

Flow rate: 1 for 6.5 min, to 1.5 over 18.5 min, maintain at 1.5 for 3 min (re-equilibrate at 1.5 mL/min)

Injection volume: 10-30

Detector: UV 207.5

CHROMATOGRAM

Retention time: 13.055

KEY WORDS

whole blood

REFERENCE

Gaillard,Y.; Pépin,G. Use of high-performance liquid chromatography with photodiode-array UV detection for the creation of a 600-compound library. Application to forensic toxicology, *J.Chromatogr.A*, **1997**, *763*, 149–163.

SAMPLE

Matrix: formulations

Sample preparation: Dissolve the contents of a 150 mg capsule in 1 L water, stir for 1 h, let stand for 30 min, centrifuge an aliquot at 1250 g for 5 min. Dilute 1 mL of the supernatant to 50 mL with water. Remove a 1 mL aliquot and add it to 100 μL 1 μg/mL IS, add 100 μL 100 mM NaOH, add 2 mL reagent, vortex, let stand for 15 min, inject a 30 μL aliquot of upper aqueous layer (sic). (Reagent was 20 μM 3,5-dinitrophenylisocyanate in dichloromethane. Prepare as follows. Stir 6.40 g 3,5-dinitrobenzoyl chloride in 100 mL glacial acetic acid, add 1.80 g sodium azide in small increments, stir for 1 h, add 300 mL cold water. Filter off the 3,5-dinitrobenzyl azide precipitate and wash it with a small portion of water. Dry overnight in a vacuum desiccator. Reflux 25 mg 3,5-dinitrobenzyl azide dissolved in 5 mL toluene for 10 min, cool to room temperature, make up to 50 mL with dichloromethane, dilute an aliquot 1:100 with dichloromethane to give a 20 μM solution of 3,5-dinitrophenylisocyanate. Prepare fresh daily. CAUTION! 3,5-Dinitrobenzyl azide may be explosive and 3,5-dinitrophenylisocyanate may be toxic!)

HPLC VARIABLES

Column: 250 × 4.6 5 μm (R)-naphthylurea Chiral (Supelco)

Mobile phase: Hexane:dichloromethane:MeOH:MeCN 81:17.5:1:0.5 (For l-isomer use 85:13.5:1:0.5 and 200 μM reagent.) (The exact ratios are very important.)

Flow rate: 1.5
Injection volume: 30
Detector: UV 235

CHROMATOGRAM
Retention time: 10.3 (d)
Internal standard: β-methylphenethylamine (17.6)
Limit of quantitation: 50 ng/mL

OTHER SUBSTANCES
Simultaneous: impurities

KEY WORDS
conduct analyses under yellow light; chiral; derivatization; capsules

REFERENCE
Dou,L.; Zeng,J.-N.; Gerochi,D.D.; Duda,M.P.; Stuting,H.H. Chiral high-performance liquid chromatography methodology for quality control monitoring of dexfenfluramine, *J.Chromatogr.A*, **1994**, *679*, 367–374.

SAMPLE
Matrix: solutions
Sample preparation: Dissolve in MeOH at a concentration of 1 mg/mL, inject a 20 μL aliquot.

HPLC VARIABLES
Column: 250 × 5 Spherisorb S5W
Mobile phase: MeOH:buffer 90:10 (Buffer was 94 mL 35% ammonia and 21.5 mL 70% nitric acid in 884 mL water, adjust the pH to 10.1 with ammonia.)
Flow rate: 2
Injection volume: 20
Detector: UV 254

CHROMATOGRAM
Retention time: 2.48

OTHER SUBSTANCES
Simultaneous: methadone, benzylmorphine, ethylmorphine, morphine-N-oxide, codeine, codeine-N-oxide, morphine, ethoheptazine, morphine-3-glucuronide, pholcodeine, norpethidine, hydrocodone, dihydrocodeine, dihydromorphine, levorphanol, norcodeine, normorphine, pemoline, benzphetamine, diethylpropion, mazindol, tranylcypromine, caffeine, fenethyline, phendimetrazine, methylphenidate, phenelzine, epinephrine, pipradol, phenylpropanolamine, fencamfamin, normetanephrine, 4-hydroxyamphetamine, bromo-STP, STP, prolintane, 2-phenethylamine, tyramine, trimethoxyamphetamine, phenylephrine, pseudoephedrine, ephedrine, methylephedrine, dimethylamphetamine, methamphetamine, mescaline, mephentermine, buprenorphine, dextromoramide, phenoperidine, fentanyl, etorphine, piritramide, noscapine, papaverine, naloxone, dextropropoxyphene, nalorphine, phenazocine, norpipanone, levallorphan, hydroxypethidine, normethadone, meperidine, dipipanone, diamorphine, pentazocine
Noninterfering: dopamine, levodopa, methyldopa, methyldopate, norepinephrine
Interfering: chlorphentermine, norpseudoephedrine, methylenedioxyamphetamine, amphetamine, acetylcodeine, monoacetylmorphine, thebacon, oxycodone, thebaine, norlevorphanol

REFERENCE
Law,B.; Gill,R.; Moffat,A.C. High-performance liquid chromatography retention data for 84 basic drugs of forensic interest on a silica column using an aqueous methanol eluent, *J.Chromatogr.*, **1984**, *301*, 165–172.

SAMPLE
Matrix: solutions
Sample preparation: Prepare a 10 μg/mL solution in MeOH, inject a 20 μL aliquot.

HPLC VARIABLES
Column: 125 × 4.9 Spherisorb S5W silica

Mobile phase: MeOH containing 10 mM ammonium perchlorate and 1 mL/L 100 mM NaOH in MeOH, pH 6.7
Flow rate: 2
Injection volume: 20
Detector: E, LeCarbone, V25 glassy carbon electrode, + 1.2 V

CHROMATOGRAM

Retention time: 2.1

OTHER SUBSTANCES

Also analyzed: acebutolol, acepromazine, acetophenazine, N-acetylprocainamide, albuterol, alprenolol, amethocaine, amiodarone, amitriptyline, antazoline, atenolol, azacyclonal, bamethan, benactyzine, benperidol, benzethidine, benzocaine, benzoctamine, benzphetamine, benzquinamide, bromhexine, bromodiphenhydramine, bromperidol, brompheniramine, brompromazine, buclizine, bufotenine, bupivacaine, buprenorphine, butacaine, butethamate, chlorcyclizine, chlorpheniramine, chlorphenoxamine, chlorprenaline, chlorpromazine, chlorprothixene, cimetidine, cinchonidine, cinnarizine, clemastine, clomipramine, clonidine, cocaine, cyclazocine, cyclizine, cyclopentamine, cyproheptadine, deserpidine, desipramine, dextromoramide, dextropropoxyphene, dicyclomine, diethylcarbamazine, diethylpropion, diethylthiambutene, dihydroergotamine, dimethindene, dimethothiazine, diphenhydramine, diphenoxylate, dipipanone, diprenorphine, dipyridamole, disopyramide, dothiepin, doxapram, doxepin, doxylamine, droperidol, ephedrine, ergocornine, ergocristine, ergocristinine, ergocryptine, ergometrine, ergosine, ergosinine, ergotamine, ethopropazine, etorphine, etoxeridine, fenethazine, fenoterol, fentanyl, flavoxate, fluopromazine, flupenthixol, fluphenazine, flurazepam, haloperidol, hydroxyzine, hyoscine, ibogaine, imipramine, indapamine, iprindole, isothipendyl, isoxsuprine, ketanserin, laudanosine, lidocaine, lofepramine, loxapine, maprotiline, mecamylamine, meclophenoxate, meclozine, medazepam, mephentermine, mepivacaine, meptazinol, mepyramine, mesoridazine, metaraminol, methadone, methamphetamine, methapyrilene, methdilazene, methotrimeprazine, methoxamine, methoxyphenamine, methoxypromazine, methylephedrine, methylergonovine, methysergide, metoclopramide, metopimazine, metoprolol, mianserin, morazone, nadolol, nalorphine, naloxone, naphazoline, nicotine, nifedipine, nomifensine, nortriptyline, noscapine, orphenadrine, oxeladin, oxprenolol, oxymetazolin, papaverine, pargyline, pecazine, penbutolol, pentazocine, penthienate, pericyazine, perphenazine, phenadoxone, phenampromide, phenazocine, phenbutrazate, phendimetrazine, phenelzine, phenglutarimide, phenindamine, pheniramine, phenmetrazine, phenomorphan, phenoperidine, phenothiazine, phenoxybenzamine, phentolamine, phenylephrine, phenyltoloxamine, physostigmine, piminodine, pimozide, pindolol, pipamazine, pipazethate, piperacetazine, piperidolate, pipradol, pirenzepine, piritramide, pizotifen, practolol, pramoxine, prazosin, prenylamine, prilocaine, primaquine, proadifen, procainamide, procaine, prochlorperazine, procyclidine, proheptazine, prolintane, promazine, promethazine, pronethalol, properidine, propiomazine, propranolol, prothipendyl, protriptyline, proxymetacaine, pseudoephedrine, pyrimethamine, quinidine, quinine, ranitidine, rescinnamine, sotalol, tacrine, terazosin, terbutaline, terfenadine, thenyldiamine, theophylline, thiethylperazine, thiopropazate, thioproperazine, thioridazine, thiothixene, thonzylamine, timolol, tocainide, tolpropamine, tolycaine, tranylcypromine, trazodone, trifluoperazine, trifluperidol, trimeperidine, trimeprazine, trimethobenzamide, trimethoprim, trimipramine, tripelennamine, triprolidine, tryptamine, verapamil, xylometazoline

REFERENCE

Jane,I.; McKinnon,A.; Flanagan,R.J. High-performance liquid chromatographic analysis of basic drugs on silica columns using non-aqueous ionic eluents. II. Application of UV, fluorescence and electrochemical oxidation detection, *J.Chromatogr.*, **1985**, *323*, 191–225.

SAMPLE

Matrix: solutions

HPLC VARIABLES

Guard column: 30 × 2.1 Spheri-5 RP-8
Column: 220 × 2.1 Spheri-5 RP-8
Mobile phase: Gradient. A was 0.08% diethylamine and 0.09% phosphoric acid in water, pH 2.3. B was MeCN:water 90:10 containing 0.08% diethylamine and 0.09% phosphoric acid. A:B 95:5 for 2 min, to 0:100 over 15 min (?), maintain at 0:100 for 5 min.
Column temperature: 50
Flow rate: 0.5

Detector: UV 200

CHROMATOGRAM
Retention time: 11.5

OTHER SUBSTANCES
Simultaneous: diethylpropion, phenylpropanolamine, ephedrine, amphetamine, methamphetamine, phentermine
Also analyzed: amitriptyline, chlordiazepoxide, chlorpromazine, desalkylflurazepam, desipramine, desmethyldoxepin, diazepam, doxepin, flurazepam, imipramine, mesoridazine, norchlordiazepoxide, nordiazepam, nortriptyline, oxazepam, prazepam, promazine, thioridazine, thiothixene, trifluoperazine

REFERENCE
Rainin Catalog, C1-94, **1994**, p. 7.24.

SAMPLE
Matrix: urine
Sample preparation: Condition a Bond Elut C8 SPE cartridge with MeOH, water, and buffer. Dilute human and dog urine with an equal volume of buffer and add to the SPE cartridge, wash with water, elute with 200 μL MeOH. Evaporate the eluate to dryness under a stream of nitrogen, reconstitute in MeOH, inject an aliquot. Inject mouse and rat urine directly. (Buffer was 100 mM pH 9.0 phosphate buffer.)

HPLC VARIABLES
Mobile phase: Gradient. A was MeOH. B was MeOH:50 mM phosphoric acid 5:95. C was MeOH water 5:95. D was MeOH:water 70:30. A:B:C:D from 30:70:0:0 to 0:0:0:100 over 20 min, to 70:30:0:0 over 5 min, maintain at 70:30:0:0 for 35 min, return to initial conditions over 1 min.
Flow rate: 1
Detector: Radioactivity or UV

CHROMATOGRAM
Retention time: 50

OTHER SUBSTANCES
Extracted: metabolites

KEY WORDS
mouse; rat; dog; human; pharmacokinetics; SPE

REFERENCE
Marchant,N.C.; Breen,M.A.; Wallace,D.; Bass,S.; Taylor,A.R.; Ings,R.M.J.; Campbell,D.B.; Williams,J. Comparative biodisposition and metabolism of ^{14}C-(±)-fenfluramine in mouse, rat, dog and man, *Xenobiotica*, **1992**, *22*, 1251–1266.

SAMPLE
Matrix: urine
Sample preparation: 1 mL Urine + 200 μL 5 M NaOH + 3 mL diethyl ether, vortex for 2 min, centrifuge at 2200 g for 5 min, freeze in acetone-dry ice. Remove the organic layer and add it to 120 μL 500 mM sulfuric acid, vortex for 2 min, centrifuge at 2200 g for 5 min. Remove the aqueous layer and evaporate traces of ether with a stream of nitrogen, inject a 100 μL aliquot.

HPLC VARIABLES
Column: 300 × 3.9 20 μm μBondapak C18
Mobile phase: MeCN:50 mM KH_2PO_4 25:75
Flow rate: 1.3
Injection volume: 100
Detector: UV 210

CHROMATOGRAM
Retention time: 14

Limit of detection: 10 ng/mL
Limit of quantitation: 25 ng/mL

OTHER SUBSTANCES
Extracted: metabolites

REFERENCE
Gross,A.S.; Phillips,A.C.; Boutagy,J.; Shenfield,G.M. Determination of dexfenfluramine and nordexfenfluramine in urine by high-performance liquid chromatography using ultraviolet detection, *J.Chromatogr.*, **1993**, *621*, 115–120.

SAMPLE
Matrix: urine
Sample preparation: 2 mL Urine + 200 μL 5 μg/mL IS in water + 400 μL water, vortex for 5 s, add 400 μL 1 M NaOH, vortex, centrifuge at 2500 g for 10 min. Remove the supernatant and add it to 5 mL hexane:ethyl acetate 70:30, shake gently by hand for 1 min, let stand for 10 min. Remove 4 mL of the upper organic layer and evaporate it to about 30 μL under a stream of nitrogen, add 1 mL reagent, vortex for 2 s, let stand for 30 min, inject a 35 μL aliquot. (Reagent was 20 μM 3,5-dinitrophenylisocyanate in dichloromethane. Prepare as follows. Stir 6.40 g 3,5-dinitrobenzoyl chloride in 100 mL glacial acetic acid, add 1.80 g sodium azide in small increments, stir for 1 h, add 300 mL cold water. Filter off the 3,5-dinitrobenzyl azide precipitate and wash it with a small portion of water. Dry overnight in a vacuum desiccator. Reflux 25 mg 3,5-dinitrobenzyl azide dissolved in 5 mL toluene for 10 min, cool to room temperature, make up to 50 mL with dichloromethane, dilute an aliquot 1:100 with dichloromethane to give a 20 μM solution of 3,5-dinitrophenylisocyanate. Prepare fresh daily. CAUTION! 3,5-Dinitrobenzyl azide may be explosive and 3,5-dinitrophenylisocyanate may be toxic!)

HPLC VARIABLES
Column: 250 × 4.6 5 μm (R)-naphthylurea Chiral (Supelco)
Mobile phase: Hexane:isopropanol:MeCN 90.5:7.5:2 (d isomer) or 89:9:2 (l isomer)
Column temperature: ambient (l isomer), 35 (d isomer)
Flow rate: 1.2
Injection volume: 35
Detector: UV 235

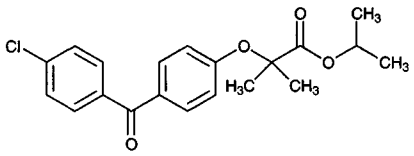
CHROMATOGRAM
Retention time: 9.3 (d isomer), 10.7 (l isomer)
Internal standard: β-methylphenethylamine (19.5, first peak, d-isomer system) (18.8, l-isomer system, first peak)
Limit of quantitation: 25 ng/mL

OTHER SUBSTANCES
Extracted: metabolites

KEY WORDS
conduct analyses under yellow light; derivatization

REFERENCE
Zeng,J.-N.; Dou,L.; Duda,M.; Stuting,H.H. New chiral high-performance liquid chromatographic methodology used for the pharmacokinetic evaluation of dexfenfluramine, *J.Chromatogr.B*, **1994**, *654*, 231–248.

Fenofibrate



Molecular formula: $C_{20}H_{21}ClO_4$
Molecular weight: 360.84
CAS Registry No.: 49562-28-9
Merck Index: 4019

SAMPLE

Matrix: blood, urine
Sample preparation: Condition a 200 mg Analytichem C8 SPE cartridge with 2 portions of MeOH and 2 portions of water, do not allow to dry. 1 mL Plasma or urine + 5 μg/mL naproxen in MeOH + 500 μL water + 250 μL 1 M HCl, mix, add to the SPE cartridge, wash with 2 portions of water, elute with 500 μL MeCN. Evaporate the eluate to 150 μL under vacuum, inject a 100 μL aliquot.

HPLC VARIABLES

Guard column: Bondapak C18/Corasil
Column: 100 × 8 10 μm Radial-Pak C8 (Waters)
Mobile phase: MeCN:buffer 35:65 (Buffer was 2.72 g KH_2PO_4 in 1 L water, pH adjusted to 3 with phosphoric acid.)
Flow rate: 2.5
Injection volume: 100
Detector: UV 254

CHROMATOGRAM

Retention time: 7 (as fenofibric acid)
Internal standard: naproxen (4.5)
Limit of quantitation: 500 ng/mL

KEY WORDS

plasma; SPE

REFERENCE

Ramusino,A.C.; Carozzi,A. Simple and rapid method for determining procetofenic acid, an active metabolite of procetofen, in biological fluids by solid-phase extraction and high-performance liquid chromatography, *J.Chromatogr.*, **1986**, *383*, 419–424.

SAMPLE

Matrix: blood, urine
Sample preparation: Add 1 mL whole blood or urine to Toxi-Tube A (Toxi-Lab, Irvine CA), add 3 mL water, mix by gentle inversion for 5 min, centrifuge at 1500 g for 5 min. Remove the organic layer and evaporate it to dryness under a stream of nitrogen at 40°, reconstitute the residue with 50 μL MeCN:water 50:50, vortex for 10 s, centrifuge at 7500 g for 2 min, inject a 10 (urine) or 30 (blood) μL aliquot. (The detector wavelength shown is the wavelength of maximum absorbance. This will not necessarily be the optimal wavelength for the separation. Multiple wavelengths from 200-350 nm can be scanned using a diode-array detector. Otherwise, 220 nm may be a reasonable choice for initial work. Matrix may interfere.)

HPLC VARIABLES

Guard column: 20 mm long Symmetry C18
Column: 250 × 4.6 5 μm Symmetry C8 (Waters)
Mobile phase: Gradient. A was 50 mM pH 3.8 sodium phosphate buffer. B was MeCN. A:B 85:15 for 6.5 min, 65:35 for 18.5 min, 20:80 for 3 min (step gradient), re-equilibrate at initial conditions for 7 min.
Column temperature: 30
Flow rate: 1 for 6.5 min, to 1.5 over 18.5 min, maintain at 1.5 for 3 min (re-equilibrate at 1.5 mL/min)
Injection volume: 10-30
Detector: UV 200.5

CHROMATOGRAM
Retention time: 18.262

KEY WORDS
whole blood

REFERENCE
Gaillard,Y.; Pépin,G. Use of high-performance liquid chromatography with photodiode-array UV detection for the creation of a 600-compound library. Application to forensic toxicology, *J.Chromatogr.A*, **1997**, *763*, 149–163.

SAMPLE
Matrix: feces, urine
Sample preparation: Urine. Add 3-5 mL urine to a C18 Sep-Pak SPE cartridge, wash with 10 mL water, elute with 10 mL MeOH. Evaporate the eluate to dryness under reduced pressure, take up the residue in 200 µL MeOH, inject an aliquot. Feces. Vortex 2 mL homogenized feces with 10 mL MeOH for 90 s, centrifuge, evaporate the supernatant to dryness under reduced pressure, take up the residue in 5 mL water, add to a C18 Sep-Pak SPE cartridge, wash with 10 mL water, elute with 10 mL MeOH. Evaporate the eluate to dryness under reduced pressure, take up the residue in 200 µL MeOH, inject an aliquot.

HPLC VARIABLES
Column: 100 × 8 RadialPAK µBondapak
Mobile phase: MeOH:water 60:40 containing 1% glacial acetic acid
Flow rate: 2.5
Detector: UV 254

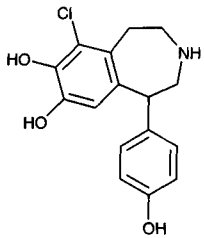
CHROMATOGRAM
Retention time: 68

OTHER SUBSTANCES
Extracted: metabolites

KEY WORDS
SPE

REFERENCE
Weil,A.; Caldwell,J.; Strolin-Benedetti,M. The metabolism and disposition of 14C-fenofibrate in human volunteers, *Drug Metab.Dispos.*, **1990**, *18*, 115–120.

Fenoldopam



Molecular formula: $C_{16}H_{16}ClNO_3$
Molecular weight: 305.76
CAS Registry No.: 67227-56-9, 67227-57-0 (monomethanesulfonate)
Merck Index: 4020
Lednicer No.: 4 147

SAMPLE
Matrix: blood
Sample preparation: Add 4.75 mL plasma to 250 µL 10% ascorbic acid before storage at -20°. 1 mL Plasma + 50 µL 500 ng/mL IS in 50 mM acetic acid + 5 mL ethyl acetate + 500 µL 0.5 mM Na_2HPO_4, shake on a reciprocal shaker at 60 cycles/min for 10 min, centrifuge at 2000 g for 10 min. Remove 4.5 mL of the organic layer and evaporate it to dryness under a stream of nitrogen at 40°, reconstitute the residue in 100 µL 200 µg/mL 2,3,4,6-tetra-O-acetyl-β-D-glucopyranosyl isothiocyanate in ethyl acetate (freshly prepared), add 50 µL 1% triethylamine in

ethyl acetate (freshly prepared), let stand at room temperature for 1 h, evaporate to dryness under a stream of nitrogen at 40°, reconstitute the residue in 200 μL MeCN:50 mM acetic acid 30:70, inject a 5-50 μL aliquot.

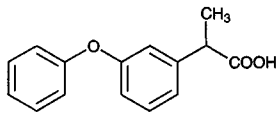
HPLC VARIABLES
Guard column: 30 × 2.1 butylsilica (Pierce)
Column: 220 × 2.1 7 μm Aquapore butylsilica (Pierce)
Mobile phase: MeCN:MeOH:buffer:water 17:15:28:42 (Prepare buffer by dissolving 22 g sodium acetate trihydrate, 21 g citric acid monohydrate, 9.8 g NaOH, and 0.63 g disodium EDTA in 2 L water, pH 5.6. Recycle mobile phase.)
Column temperature: 37
Flow rate: 0.3
Injection volume: 5-50
Detector: E, ESA, guard electrode +0.2 V, working electrode 1 -0.20 V, working electrode 2 +0.20 V

CHROMATOGRAM
Retention time: 3 (S), 6 (R)
Internal standard: 2,3,4,5-tetrahydro-1-phenyl-1H-3-benzazepine-7,8-diol (SK&F 38393-A) (25, 30 (enantiomers))
Limit of detection: 0.25 ng/mL
Limit of quantitation: 0.5 ng/mL

KEY WORDS
derivatization; chiral; plasma

REFERENCE
Boppana,V.K.; Geschwindt,L.; Cyronak,M.J.; Rhodes,G. Determination of the enantiomers of fenoldopam in human plasma by reversed-phase high-performance liquid chromatography after chiral derivatization, *J.Chromatogr.*, **1992**, *592*, 317–322.

Fenoprofen



Molecular formula: $C_{15}H_{14}O_3$
Molecular weight: 242.27
CAS Registry No.: 31879-05-7, 34507-40-5 (calcium salt), 53746-45-5 (calcium salt dihydrate)
Merck Index: 4021
Lednicer No.: 2 67

SAMPLE
Matrix: blood
Sample preparation: Activate a 1 mL Bond-Elut C8 SPE cartridge with 2 mL MeOH then 1 mL 10 mM HCl, do not allow it to dry completely. Sonicate 1 mL whole blood for 20-30 min then apply to cartridge. Wash with 100 μL water, elute with three 500 μL portions of MeOH:MeCN:1% aqueous ammonium hydroxide 50:20:30, combine eluents and evaporate to dryness under a stream of nitrogen at 40°. Redissolve in 1 mL MeOH, inject a 20 μL aliquot.

HPLC VARIABLES
Column: 250 × 4.5 5 μm Spherisorb ODS
Mobile phase: MeCN:MeOH:buffer 35:13:52 (Buffer was water adjusted to pH 3.2 with ortho-phosphoric acid
Flow rate: 1
Injection volume: 20
Detector: UV 250

CHROMATOGRAM
Retention time: 6.5

OTHER SUBSTANCES
Simultaneous: ketoprofen, acetaminophen, salicylic acid, naproxen, ibuprofen, indomethacin

KEY WORDS
whole blood; SPE

REFERENCE
Moore,C.M.; Tebbett,I.R. Rapid extraction of anti-inflammatory drugs in whole blood for HPLC analysis, *Forensic Sci.Int.*, **1987**, *34*, 155–158.

SAMPLE
Matrix: blood
Sample preparation: 500 μL Plasma + 200 μL 2 M HCl + 6 mL ice-cold hexane:diethyl ether 8:2, extract, centrifuge at 1500 g for 10 min. Remove 5 mL of organic layer and evaporate it to dryness under a stream of nitrogen. Dissolve in 250 μL isopropanol:water 2:8, inject a 40 μL aliquot.

HPLC VARIABLES
Column: 100 × 4.0 AGP (EnantioPac)
Mobile phase: 20 mM pH 6.7 phosphate buffer containing 0.5% isopropanol and 5 mM dimethyloctylamine
Column temperature: 15
Flow rate: 0.5
Injection volume: 40
Detector: UV 220

CHROMATOGRAM
Retention time: 34 (R), 42 (S)
Limit of quantitation: 250 ng/mL

OTHER SUBSTANCES
Simultaneous: ibuprofen

KEY WORDS
plasma; chiral

REFERENCE
Menzel-Soglowek,S.; Geisslinger,G.; Brune,K. Stereoselective high-performance liquid chromatographic determination of ketoprofen, ibuprofen and fenoprofen in plasma using a chiral α_1-acid glycoprotein column, *J.Chromatogr.*, **1990**, *532*, 295–303.

SAMPLE
Matrix: blood
Sample preparation: 500 μL Plasma + 50 μL MeOH:water 1:4 + 200 μL 1 M sulfuric acid + 3 mL isooctane:isopropanol 95:5, vortex 30 s, centrifuge at 1800 g for 5 min. Remove organic layer and evaporate it to dryness. Add 300 μL 50 mM triethylamine in MeCN and 50 μL 6 mM ethylchloroformate in MeCN, wait 30 s, add 25 μL 0.1% (S)-naphthylethylamine in MeCN: triethylamine 98:2, after 3 min add 25 μL 2.5% ethanolamine in MeCN, inject 2-30 μL aliquot.

HPLC VARIABLES
Column: 100 × 4.6 5 μm Partisil ODS 3 RAC
Mobile phase: MeCN:water:acetic acid:triethylamine 60:40:0.1:0.02, final pH 5.0 (After every third injection flush with MeCN for 6 min at 1.6 mL/min, equilibrate with mobile phase for 9 min.)
Flow rate: 1.2
Injection volume: 2-30
Detector: F ex 280 em 320

CHROMATOGRAM
Retention time: 7.5 (S), 8.8 (R)
Internal standard: fenoprofen

Limit of detection: 10 ng/mL

OTHER SUBSTANCES
Extracted: ibuprofen

KEY WORDS
plasma; chiral; UV 232 (see Clin.Chem. 1988; 34; 493); derivatization; fenoprofen is IS

REFERENCE
Lemko,C.H.; Caillé,G.; Foster,R.T. Stereospecific high-performance liquid chromatographic assay of ibuprofen: improved sensitivity and sample processing efficiency, *J.Chromatogr.*, **1993**, *619*, 330–335.

SAMPLE
Matrix: blood
Sample preparation: 500 μL Plasma + 125 μL 40 mM decanoic acid in MeCN, mix. Dialyze a 100 μL sample against 20 mM pH 7.0 phosphate buffer using a Gilson Cuprophane membrane (molecular mass cut-off 15 kDa). Continuously pump the buffer through the dialysis cell and through column A at 3 mL/min for 9.6 min, backflush the contents of column A onto column B with the mobile phase, monitor the effluent from column B. (After each injection flush plasma channel with 1 mL 0.05% Triton X-100, with 1 mL 1 mM HCl, and with 2 mL water. After each injection flush buffer channel with 3 mL 20 mM pH 7.0 phosphate buffer and condition column A with 1 mL 20 mM pH 7.0 phosphate buffer.)

HPLC VARIABLES
Column: A 10 × 2 40 μm Bondesil C18 (Analytichem); B 250 × 3.1 5 μm C18 (RoSil Research Separation Laboratories)
Mobile phase: MeCN:MeOH:20 mM pH 3.2 phosphate buffer 50:10:40
Flow rate: 1
Injection volume: 100
Detector: UV 272

CHROMATOGRAM
Limit of detection: 500 ng/mL

OTHER SUBSTANCES
Extracted: flurbiprofen (UV 247), ibuprofen (UV 264), ketoprofen (UV 261), naproxen (UV 272)

KEY WORDS
plasma; dialysis; column-switching

REFERENCE
Herráez-Hernández,R.; Van de Merbel,N.C.; Brinkman,U.A.T. Determination of the total concentration of highly protein-bound drugs in plasma by on-line dialysis and column liquid chromatography: application to non-steroidal anti-inflammatory drugs, *J.Chromatogr.B*, **1995**, *666*, 127–137.

SAMPLE
Matrix: blood
Sample preparation: 2 mL Whole blood or plasma + 2 mL buffer + 5 mL chloroform:isopropanol: n-heptane 60:14:26, shake gently horizontally for 10 min, centrifuge at 2800 g for 10 min. Remove the lower organic layer and evaporate it to dryness under vacuum at 45°, reconstitute the residue in 100 μL mobile phase, centrifuge at 2800 g for 5 min, inject a 50 μL aliquot of the supernatant. (Buffer was saturated ammonium chloride solution 25% diluted with water, adjusted to pH 9.5 with 25% ammonia solution.)

HPLC VARIABLES
Column: 300 × 3.9 4 μm NovaPack C18
Mobile phase: MeOH:THF:buffer 65:5:30 (Buffer was 0.68 g/L (10 mM (sic)) KH_2PO_4 adjusted to pH 2.6 with concentrated orthophosphoric acid.) (At the end of each session wash the column with water for 1 h and MeOH for 1 h, re-equilibrate for 30 min.)
Column temperature: 30
Flow rate: 0.8

Injection volume: 50
Detector: UV 272

CHROMATOGRAM
Retention time: 7.20
Limit of detection: <120 ng/mL

KEY WORDS
whole blood; plasma; interferences may occur–compounds (all of which are extracted) elute in this order tenoxicam; iproniazid; methocarbamol; methotrexate; caffeine; nialamide; colchicine; cytarabine; benzoylecgonine; acetaminophen; diazoxide; dacarbazine; sulfinpyrazole; flumazenil; sulpride; morphine; atenolol; toloxatone; terbutaline; albuterol; phenobarbital; ranitidine; tiapride; phenol; chlormezanone; aspirin; metformin; ritodrine; codeine; sultopride; amisulpride; naltrexone; lisinopril; benzocaine; nizatidine; nalorphine; mephenesin; naloxone; sotalol; carteolol; procainamide; carbamazepine; bromazepam; nalbuphine; nadolol; procarbazine; dihydralazine; omeprazole; strychnine; acebutolol; glutethimide; chlorpropamide; glipizide; triazolam; prazosin; flunitrazepam; clonazepam; metoclopramide; melphalan; estazolam; tolbutamide; ephedrine; clonidine; pindolol; clobazam; minoxidil; disopyramide; nitrazepam; dextromethorphan; tofisopam; zopiclone; debrisoquine; sulindac; alprazolam; cycloguanil; lorazepam; methaqualone; ketamine; piroxicam; metoprolol; nifedipine; quinine; mephentermine; prilocaine; pentazocine; oxazepam; tiaprofenic acid; quinidine; celiprolol; ajmaline; yohimbine; lidocaine; secobarbital; viloxazine; mepivacaine; meperidine; doxylamine; labetalol; temazepam; amodiaquine; benperidol; droperidol; hydroxychloroquine; zolpidem; ketoprofen; alminoprofen; cicletanine; moclobemide; chloroquine; cocaine; timolol; nomifensine; ticlopidine; acenocoumarol; vindesine; mexiletine; dipyridamole; trazodone; pipamperone; pyrimethamine; benazepril; vincristine; metapramine; chlordiazepoxide; oxprenolol; warfarin; clorazepate; flecainide; phencyclidine; thiopental; fenfluramine; metipranolol; triprolidine; naproxen; buprenorphine; verapamil; buspirone; tianeptine; midazolam; bupivacaine; carbinoxamine; loprazolam; cetirizine; chlorpheniramine; moperone; cibenzoline; medifoxamine; astemizole; vinblastine; nicardipine; bisoprolol; diltiazem; glibornuride; reserpine; aconitine; nitrendipine; diazepam; mianserin; ramipril; haloperidol; tetracaine; alprenolol; aceprometazine; glibenclamide; chlorophenacinone; doxepin; nimodipine; diphenhydramine; cyclizine; histapyrrodine; phenylbutazone; demexiptiline; clozapine; proguanil; trifluperidol; medazepam; cyamemazine; bumadizone; suriclone; propranolol; acepromazine; dothiepin; dextromoramide; fenoprofen; dextropropoxyphene; loxapine; betaxolol; propafenone; promethazine; thioproperazine; methadone; amoxapine; quinupramine; opipramol; cyproheptadine; brompheniramine; mefenidramine; protriptyline; flurbiprofen; tetrazepam; zorubicin; prazepam; alimemazine; loperamide; imipramine; desipramine; levomepromazine; hydroxyzine; niflumic acid; penbutolol; fluvoxamine; pimozide; daunorubicin; indomethacin; maprotiline; tropatenine; etodolac; fluoxetine; amitriptyline; nortriptyline; tioclomarol; diclofenac; mefloquine; trimipramine; chlorambucil; lidoflazine; ibuprofen; floctafenine; alpidem; loratadine; chlorpromazine; clomipramine; carpipramine; thioridazine; fentiazac; clemastine; mefenamic acid; fluphenazine; prochlorperazine; penfluridol; bepridil; terfenadine; trifluoperazine

REFERENCE
Tracqui,A.; Kintz,P.; Mangin,P. Systematic toxicological analysis using HPLC/DAD, *J.Forensic Sci.*, **1995**, *40*, 254–262.

SAMPLE
Matrix: blood
Sample preparation: 500 μL Plasma + 50 μL water + 200 μL 20% sulfuric acid + 6 mL n-butyl chloride, vortex for 5 min, centrifuge at 950 g for 5 min, freeze in dry ice/acetone. Remove the organic layer and evaporate it to dryness under a stream of nitrogen, reconstitute the residue in 300 μL 50 mM triethylamine, sonicate for 1 min, vortex for 30 s, add 50 μL 6 mM ethyl chloroformate, let stand for 30 s, add 25 μL 10 mM S-(-)-1-(1-naphthyl)ethylamine, let stand for 3 min, add 25 μL MeCN:ethanolamine 40:1. Evaporate to dryness under a stream of nitrogen at 40°, reconstitute the residue in 250 μL mobile phase, inject a 25 μL aliquot.

HPLC VARIABLES
Guard column: 15 × 3.2 7 μm Newguard RP-18
Column: 150 × 4.6 5 μm Inertsil ODS-2
Mobile phase: MeCN:water (pH 3.0) 66.5:33.5
Column temperature: 27

Flow rate: 1.2
Injection volume: 25
Detector: F ex 280 em 320

CHROMATOGRAM
Retention time: 7.7 (S), 8.5 (R)
Internal standard: fenoprofen

OTHER SUBSTANCES
Extracted: ibuprofen

KEY WORDS
derivatization; chiral; plasma; fenoprofen is IS

REFERENCE
Lau,Y.Y. Determination of ibuprofen enantiomers in human plasma by derivatization and high-performance liquid chromatography with fluorescence detection, *J.Liq.Chromatogr.Rel.Technol.*, **1996**, *19*, 2143–2153.

SAMPLE
Matrix: blood, urine
Sample preparation: Plasma. 500 μL Plasma + 50 μL 50 μg/mL ketoprofen in 10 mM NaOH + 100 μL 600 mM sulfuric acid + 3 mL isooctane:isopropanol 95:5, vortex for 30 s, centrifuge at 3000 rpm for 5 min. Remove the organic layer and add it to 2.5 mL water, vortex for 15 s, centrifuge for 3 min. Remove the aqueous layer and add it to 200 μL 600 mM sulfuric acid, add 2.5 mL chloroform, vortex for 15 s, centrifuge for 3 min. Remove the organic layer and evaporate it to dryness under reduced pressure, reconstitute with 100 μL 50 mM triethylamine in MeCN, add 50 μL 60 mM ethyl chloroformate in MeCN, after 30 s add 50 μL 1 M l-leucinamide in MeOH containing 1 M triethylamine, after 2 min add 50 μL water, inject a 10-40 μL aliquot. Urine. 500 μL Urine + 250 μL 1 M NaOH, mix, add 300 μL 600 mM sulfuric acid, add 50 μL 50 μg/mL ketoprofen in 10 mM NaOH, add 3 mL isooctane:isopropanol 95:5, vortex for 30 s, centrifuge at 3000 rpm for 5 min. Remove the organic layer and add it to 2.5 mL water, vortex for 15 s, centrifuge for 3 min. Remove the aqueous layer and add it to 200 μL 600 mM sulfuric acid, add 2.5 mL chloroform, vortex for 15 s, centrifuge for 3 min. Remove the organic layer and evaporate it to dryness under reduced pressure, reconstitute with 100 μL 50 mM triethylamine in MeCN, add 50 μL 60 mM ethyl chloroformate in MeCN, after 30 s add 50 μL 1 M l-leucinamide in MeOH containing 1 M triethylamine, after 2 min add 50 μL water, inject a 10-40 μL aliquot.

HPLC VARIABLES
Guard column: 20 mm long 37-53 μm reverse phase
Column: 100 × 4.6 5 μm Partisil 5 ODS-3
Mobile phase: MeCN:70 mM KH_2PO_4:triethylamine 65:35:0.02, pH 6.0
Flow rate: 1 (plasma), 1.2 (urine)
Injection volume: 10-40
Detector: UV 275 for 13 min then UV 232

CHROMATOGRAM
Retention time: 16.3 (R, urine), 19.1 (R, plasma or S, urine), 22.0 (S, plasma)
Internal standard: ketoprofen (9, 10 (enantiomers))
Limit of quantitation: 250 ng/mL

KEY WORDS
plasma; derivatization; pharmacokinetics; chiral

REFERENCE
Mehvar,R.; Jamali,F. Stereospecific high-performance liquid chromatographic (HPLC) assay of fenoprofen enantiomers in plasma and urine, *Pharm.Res.*, **1988**, *5*, 53–56.

SAMPLE
Matrix: blood, urine

Sample preparation: Plasma. Adjust pH of 1 mL of plasma immediately to 2-4 with 50 μL 85% phosphoric acid, add 2 mL MeCN, centrifuge, evaporate the MeCN, extract with 3 mL ethyl acetate. Evaporate the organic solvent to dryness, reconstitute in 250 μL mobile phase, 10 μL 0.125 mM ketoprofen, and 20 μL 0.2 mM flunoxaprofen, inject a 100 μL aliquot. Urine. 500 μL Urine + 500 μL mobile phase + 50 μL 0.25 mM ketoprofen, inject a 100 μL aliquot.

HPLC VARIABLES
Column: 250 × 4.6 5 μm Ultrasphere ODS
Mobile phase: Gradient. MeCN:10 mM pH 2.5 tetrabutylammonium hydrogen sulfate 25:75 for 15 min, 32:68 for 10 min, 38:62 for 12 min (step gradient).
Flow rate: 1
Injection volume: 100
Detector: UV 272

CHROMATOGRAM
Retention time: 45.4
Internal standard: ketoprofen (29.9), flunoxaprofen (42.2)
Limit of detection: 100 ng/mL
Limit of quantitation: 250 ng/mL

OTHER SUBSTANCES
Extracted: metabolites, glucuronides

KEY WORDS
plasma; pharmacokinetics

REFERENCE
Volland,C.; Sun,H.; Benet,L.Z. Stereoselective analysis of fenoprofen and its metabolites, *J.Chromatogr.*, **1990**, *534*, 127–138.

SAMPLE
Matrix: blood, urine
Sample preparation: 100 μL Urine or rat plasma or 500 μL human plasma + 50 μL MeOH:10 mM NaOH 10:90 + 200 μL 600 mM sulfuric acid + 3 mL isooctane:isopropanol 95:5, vortex for 30 s, centrifuge at 1800 g for 5 min. Remove the organic layer and evaporate it to dryness, reconstitute the residue in 200 μL 50 mM triethylamine in MeCN, add 50 μL 6 mM ethyl chloroformate in MeCN, vortex for 30 s, add 50 μL 500 mM R-(+)-α-phenylethylamine in MeCN: triethylamine 80:20, vortex briefly, let stand for 2 min, add 1 mL 250 mM HCl, add 3 mL chloroform, vortex for 30 s, centrifuge at 1800 g for 2 min. Remove the organic layer and evaporate it to dryness, reconstitute the residue in 200 μL mobile phase, inject a 10-150 μL aliquot.

HPLC VARIABLES
Guard column: 20 × 4.6 37-53 μm reversed-phase
Column: 100 × 4.6 5 μm C18 (Phenomenex)
Mobile phase: MeCN:water:acetic acid:triethylamine 46.5:53.5:0.1:0.03, pH 4.9
Flow rate: 1.6
Injection volume: 10-150
Detector: UV 225

CHROMATOGRAM
Retention time: 11.70, 13.40 (enantiomers)
Internal standard: fenoprofen

OTHER SUBSTANCES
Extracted: ibuprofen

KEY WORDS
derivatization; human; chiral; rat; fenoprofen is IS; plasma

REFERENCE
Wright,M.R.; Sattari,S.; Brocks,D.R.; Jamali,F. Improved high-performance liquid chromatographic assay method for the enantiomers of ibuprofen, *J.Chromatogr.*, **1992**, *583*, 259–265.

SAMPLE
Matrix: blood, urine
Sample preparation: Plasma. 500 μL Plasma + 100 μL 600 mM sulfuric acid + 4 mL 2,2,4-trimethylpentane:isopropanol 95:5, vortex for 10 s, centrifuge at 1800 g for 5 min. Remove the organic layer and evaporate it to dryness under reduced pressure, reconstitute the residue in 180 μL mobile phase, vortex for 10 s, inject a 100 μL aliquot. Urine. 500 μL Urine + 100 μL 600 mM sulfuric acid + 4 mL 2,2,4-trimethylpentane:isopropanol 95:5, vortex for 10 s, centrifuge at 1800 g for 3 min. Remove the organic layer and add it to 3 mL water, vortex for 10 s, centrifuge for 3 min. Remove the aqueous phase and add it to 200 μL 600 mM sulfuric acid and 3 mL chloroform, vortex for 10 s, centrifuge for 3 min. Remove the organic phase and evaporate it to dryness, reconstitute the residue in 180 μL mobile phase, vortex for 10 s, inject a 100 μL aliquot.

HPLC VARIABLES
Column: 250 × 4.6 Chiralpak AD amylose carbamate (Chiral Technologies)
Mobile phase: Hexane:isopropanol:trifluoroacetic acid 80:19.9:0.1
Flow rate: 1
Injection volume: 100
Detector: UV 254

CHROMATOGRAM
Retention time: 5.3, 6.3 (enantiomers)
Internal standard: fenoprofen

OTHER SUBSTANCES
Extracted: ketoprofen

KEY WORDS
plasma; chiral; fenoprofen is IS

REFERENCE
Carr,R.A.; Caillé,G.; Ngoc,A.H.; Foster,R.T. Stereospecific high-performance liquid chromatographic assay of ketoprofen in human plasma and urine, *J.Chromatogr.B*, **1995**, *668*, 175–181.

SAMPLE
Matrix: blood, urine
Sample preparation: Plasma. 2 mL Plasma + 250 μL 1 M HCl + 50 μL prenazone solution, vortex briefly, extract twice with 5 mL portions of diethyl ether for 15 min each time. Combine the organic layers and evaporate them to dryness under a stream of nitrogen at 36°, reconstitute the residue in 200 μL MeCN:1% aqueous acetic acid 50:50, inject an aliquot. Urine. 500 μL Urine + 250 μL 1 M HCl + 50 μL prenazone solution, mix, extract with 5 mL diethyl ether for 15 min. Remove the organic layer and add it to 1 mL 1% sodium bicarbonate solution (freshly prepared), vortex. Remove the organic layer and evaporate it to dryness under a stream of nitrogen at 36°, reconstitute the residue in 200 μL MeCN:1% aqueous acetic acid 50:50, inject an aliquot. (To hydrolyze conjugates mix 500 μL urine, 100 μL 1 M pH 5.2 sodium acetate buffer, and 20 μL enzyme solution, heat at 56° for 2.5 h, cool, proceed as above. The enzyme solution was Suc Helix Pomatia containing 100 000 Fishman U/mL of β-glucuronidase and 1 000 000 Roy U/mL of arylsulfatase (IBF).)

HPLC VARIABLES
Column: 100 × 3 5 μm Nucleosil
Mobile phase: Gradient. MeCN:1% aqueous acetic acid 50:50 for 7 min, to 80:20 over 0.6 min, maintain at 80:20 for 3.4 min, re-equilibrate at initial conditions for 8 min. (For urine isocratic MeCN:1% aqueous acetic acid 50:50.)
Flow rate: 0.5
Injection volume: 20
Detector: UV 230

CHROMATOGRAM
Internal standard: prenazone
Limit of quantitation: 200 ng/mL (urine), 50 ng/mL (plasma)

KEY WORDS
horse; plasma; pharmacokinetics

REFERENCE
Delbeke,F.T.; Landuyt,J.; Debackere,M. Disposition of human drug preparations in the horse. IV. Orally administered fenoprofen, *J.Pharm.Biomed.Anal.*, **1995**, *13*, 1041–1047.

SAMPLE
Matrix: blood, urine
Sample preparation: Add 1 mL whole blood or urine to Toxi-Tube A (Toxi-Lab, Irvine CA), add 3 mL water, mix by gentle inversion for 5 min, centrifuge at 1500 g for 5 min. Remove the organic layer and evaporate it to dryness under a stream of nitrogen at 40°, reconstitute the residue with 50 μL MeCN:water 50:50, vortex for 10 s, centrifuge at 7500 g for 2 min, inject a 10 (urine) or 30 (blood) μL aliquot. (The detector wavelength shown is the wavelength of maximum absorbance. This will not necessarily be the optimal wavelength for the separation. Multiple wavelengths from 200-350 nm can be scanned using a diode-array detector. Otherwise, 220 nm may be a reasonable choice for initial work. Matrix may interfere.)

HPLC VARIABLES
Guard column: 20 mm long Symmetry C18
Column: 250 × 4.6 5 μm Symmetry C8 (Waters)
Mobile phase: Gradient. A was 50 mM pH 3.8 sodium phosphate buffer. B was MeCN. A:B 85:15 for 6.5 min, 65:35 for 18.5 min, 20:80 for 3 min (step gradient), re-equilibrate at initial conditions for 7 min.
Column temperature: 30
Flow rate: 1 for 6.5 min, to 1.5 over 18.5 min, maintain at 1.5 for 3 min (re-equilibrate at 1.5 mL/min)
Injection volume: 10-30
Detector: UV 200.5

CHROMATOGRAM
Retention time: 21.16

KEY WORDS
whole blood

REFERENCE
Gaillard,Y.; Pépin,G. Use of high-performance liquid chromatography with photodiode-array UV detection for the creation of a 600-compound library. Application to forensic toxicology, *J.Chromatogr.A*, **1997**, *763*, 149–163.

SAMPLE
Matrix: bulk
Sample preparation: 10 mg Compound + 10 mg 1-(3-dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride + 2 drops 3,5-dimethylaniline + 1.5 mL dichloromethane, mix, after 30 min add 1 mL 1 M HCl, shake vigorously. Remove the lower organic layer and dry it over anhydrous magnesium sulfate, inject an aliquot.

HPLC VARIABLES
Column: 250 × 4.6 5 μm D N-(3,5-dinitrobenzoyl)phenylglycine (Regis)
Mobile phase: Hexane:isopropanol 80:20
Flow rate: 2
Injection volume: 20
Detector: UV 254, UV 280

CHROMATOGRAM
Retention time: k' 2.50 (for first enantiomer)

OTHER SUBSTANCES
Also analyzed: carprofen, cicloprofen, etodolac, flurbiprofen, ibuprofen, ketoprofen, naproxen, pirprofen, tiaprofenic acid

KEY WORDS
derivatization; α = 1.25; chiral

REFERENCE
Pirkle,W.H.; Murray,P.G. The separation of the enantiomers of a variety of non-steroidal anti-inflammatory drugs (NSAIDS) as their anilide derivatives using a chiral stationary phase, *J.Liq.Chromatogr.*, **1990**, *13*, 2123–2134.

SAMPLE
Matrix: cell suspensions
Sample preparation: Centrifuge cell suspension at 2000 g for 4 min. Remove a 2 mL aliquot of the supernatant and add it to 200 µL 100 µg/mL IS in DMF, mix, add 200 µL 5 M HCl, extract twice with 3 mL portions of toluene. Combine the organic layers and evaporate them to dryness under a stream of nitrogen, reconstitute the residue in 1 mL dichloromethane, add 20 µL 10 mg/mL 1-hydroxybenzotriazole in dichloromethane:pyridine 99:1, add 300 µL 10 mg/mL 1-(3-dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride in dichloromethane, add 300 µL 10 mg/mL (-)-(S)-α-methylbenzylamine in dichloromethane, let stand for 30 min, evaporate to dryness, reconstitute with 500 µL mobile phase, inject a 10 µL aliquot.

HPLC VARIABLES
Guard column: 10 mm long Techsphere ODS (HPLC Technology, Macclesfield UK)
Column: 250 × 5 5 µm Techsphere ODS (HPLC Technology, Macclesfield UK)
Mobile phase: MeCN:7.5 mM NaH_2PO_4 55:45, containing 5 mM sodium pentanesulfonate, pH adjusted to 2.8 with phosphoric acid
Flow rate: 1
Injection volume: 10
Detector: UV 254

CHROMATOGRAM
Retention time: k' 4.95, 5.42 (enantiomers)
Internal standard: (S)-naproxen (100 µg/mL)
Limit of detection: 5 µg/mL

KEY WORDS
derivatization; chiral

REFERENCE
Thomason,M.J.; Hung,Y.-F.; Rhys-Williams,W.; Hanlon,G.W.; Lloyd,A.W. Indirect enantiomeric separation of 2-arylpropionic acids and structurally related compounds by reversed phase HPLC, *J.Pharm.Biomed.Anal.*, **1997**, *15*, 1765–1774.

SAMPLE
Matrix: solutions

HPLC VARIABLES
Guard column: 4 × 4 5 µm LiChroCart LiChrospher 100 RP-18
Column: 125 × 4 5 µm LiChroCart LiChrospher 100 RP-18
Mobile phase: MeCN:pH 4.8 sodium acetate 38:62
Flow rate: 1.5
Injection volume: 50
Detector: UV 223

CHROMATOGRAM
Internal standard: fenoprofen

OTHER SUBSTANCES
Simultaneous: ibuprofen

REFERENCE

Dominkus,M.; Nicolakis,M.; Kotz,R.; Wilkinson,F.E.; Kaiser,R.R.; Chlud,K. Comparison of tissue and plasma levels of ibuprofen after oral and topical administration, *Arzneimittelforschung*, **1997**, *46*, 1138–1143.

SAMPLE

Matrix: solutions

HPLC VARIABLES

Column: 250 × 4.6 Zorbax RX

Mobile phase: Gradient. A was 10 mL concentrated orthophosphoric acid and 7 mL triethylamine in 1 L water. B was 10 mL concentrated orthophosphoric acid and 7 mL triethylamine in 200 mL water, make up to 1 L with MeCN. A:B from 100:0 to 0:100 over 30 min, maintain at 0:100 for 5 min.

Column temperature: 30

Flow rate: 2

Detector: UV 210

OTHER SUBSTANCES

Also analyzed: acepromazine, acetaminophen, acetophenazine, albuterol, aminophylline, amitriptyline, amobarbital, amoxapine, amphetamine, amylocaine, antipyrine, aprobarbital, aspirin, atenolol, atropine, avermectin, barbital, benzocaine, benzoic acid, benzotropine, benzphetamine, berberine, bibucaine, bromazepan, brompheniramine, buprenorphine, buspirone, butabarbital, butacaine, butethal, caffeine, carbamazepine, carbromal, chloramphenicol, chlordiazepoxide, chloroquine, chlorothiazide, chloroxylenol, chlorphenesin, chlorpheniramine, chlorpromazine, chlorpropamide, chlortetracycline, cimetidine, cinchonidine, cinchonine, clenbuterol, clonazepam, clonixin, clorazepate, cocaine, codeine, colchicine, cortisone, coumarin, cyclazocine, cyclobenzaprine, cyclothiazide, cyheptamide, cymarin, danazol, danthron, dapsone, debrisoquine, desipramine, dexamethasone, dextromethorphan, dextropropoxyphene, diamorphine, diazepam, diclofenac, diethylpropion, diethylstilbestrol, diflunisal, digitoxin, digoxin, diltiazem, diphenhydramine, diphenoxylate, diprenorphine, dipyrone, disulfiram, dopamine, doxapram, doxepin, dronabinol, ephedrine, epinephrine, epinine, estradiol, estriol, estrone, ethacrynic acid, ethosuximide, etonitazene, etorphine, eugenol, famotidine, fenbendazole, fenproporex, fentanyl, flubendazole, flufenamic acid, flunitrazepam, 5-fluorouracil, fluoxymesterone, fluphenazine, furosemide, gentisic acid, gitoxigenin, glipizide, glunixin, glutethimide, glybenclamide, guaiacol, halazepam, haloperidol, hydrochlorothiazide, hydrocodone, hydrocortisone, hydromorphone, hydroxyquinoline, ibogaine, ibuprofen, iminostilbene, imipramine, indomethacin, isocarbostyril, isocarboxazid, isoniazid, isoproterenol, isoxsuprine, ivermectin, ketamine, ketoprofen, kynurenic acid, levorphanol, lidocaine, lorazepam, lormetazepam, loxapine, mazindol, mebendazole, meclizine, meclofenamic acid, medazepam, mefenamic acid, megestrol, mepacrine, meperidine, mephentermine, mephenytoin, mephesin, mephobarbital, mepivacaine, mescaline, mesoridazine, methadone, methamphetamine, methapyrilene, methaqualone, methazolamide, methocarbamol, methoxamine, methsuximide, methyl salicylate, methyldopa, methyldopamine, methylphenidate, methylprednisolone, methyltestosterone, methyprylon, metoprolol, mibolerone, morphine, nadolol, nalorphine, naloxone, naltrexone, naphazoline, naproxen, nefopam, niacinamide, nicotine, niacin, nifedipine, niflumic acid, nitrazepam, norepinephrine, nortriptyline, noscapine, nylidrin, oxazepam, oxycodone, oxymorphone, oxyphenbutazone, oxytetracycline, papaverine, pargyline, pemoline, pentazocine, pentobarbital, persantine, phenacetin, phenazocine, phenazopyridine, phencyclidine, phendimetrazine, phenelzine, pheniramine, phenobarbital, phenothiazine, phensuximide, phentermine, phenylbutazone, phenylephrine, phenylpropanolamine, piperocaine, prazepam, prednisolone, primidone, probenecid, progesterone, propiomazine, propranolol, propylparaben, pseudoephedrine, puromycin, pyrilamine, pyrithyldione, quazepam, quinaldic acid, quinidine, quinine, ranitidine, recinnamine, reserpine, resorcinol, saccharin, albuterol, salicylamide, salicylic acid, scopolamine, scopoletin, secobarbital, strychnine, sulfacetamide, sufadiazine, sulfadimethoxine, sulfaethidole, sulfamerazine, sulfamethazine, sulfamethoxizole, sulfanilamide, sulfapyridine, sulfasoxizole, sulindac, tamoxifen, temazepam, testosterone, tetracaine, tetracycline, tetramisole, thebaine, theobromine, theophylline, thiabendazole, thiamine, thiamylal, thiobarbituric acid, thioridazine, thiosalicylic acid, thiothixene, thymol, tolazamide, tolazoline, tobutamide, tolmetin, tranylcypromine, triamcinolone, tribenzylamine, trichloromethiazide, trifluoperazine, trihexyphenidyl, trimethoprim, tripelennamine, triprolidine, tropacocaine, tyramine, verapamil, vincamine, warfarin, yohimbine, zoxazolamine

REFERENCE
Hill,D.W.; Kind,A.J. Reversed-phase solvent gradient HPLC retention indexes of drugs, *J.Anal.Toxicol.*, **1994**, *18*, 233–242.

SAMPLE
Matrix: solutions
Sample preparation: Mix 1 mL 100 μg/mL compound in dichloromethane with 300 μL 100 μg/mL 1-hydroxybenzotriazole in dichloromethane:pyridine 99:1, 300 μL 1.1 mg/mL 1-ethyl-3-dimethylaminopropylcarbodiimide hydrochloride in dichloromethane, and 300 μL 300 μg/mL benzylamine in dichloromethane, vortex, let stand at room temperature for 1.5 h, evaporate to dryness under a stream of nitrogen, reconstitute the residue in 500 μL MeOH, inject an aliquot.

HPLC VARIABLES
Column: 150 × 4.6 10 μm EXP B101 tris(4-methylbenzoate) cellulose on silica (Bio-Rad)
Mobile phase: MeOH:buffer 70:30 (Prepare buffer solution by dissolving 14.05 g sodium perchlorate in water, adjust pH to 2.0, make up to 1 L with water.)
Flow rate: 1
Detector: UV 230

CHROMATOGRAM
Retention time: 14, 21 (enantiomers)

OTHER SUBSTANCES
Also analyzed: benoxaprofen (MeOH:buffer 80:20), carprofen, flurbiprofen, ibuprofen, ketoprofen, pirprofen, tiaprofenic acid

KEY WORDS
derivatization; chiral

REFERENCE
Van Overbeke,A.; Baeyens,W.; Van den Bossche,W.; Dewaele,C. Separation of 2-arylpropionic acids on a cellulose based chiral stationary phase by RP-HPLC, *J.Pharm.Biomed.Anal.*, **1994**, *12*, 901–909.

SAMPLE
Matrix: solutions

HPLC VARIABLES
Column: 125 × 3 Ecocart LiChrospher 100 RP-18
Mobile phase: MeCN:100 mM pH 7.4 KH_2PO_4 30:70
Flow rate: 0.4
Injection volume: 2.5
Detector: F ex 271 em 293

CHROMATOGRAM
Retention time: 4.6

OTHER SUBSTANCES
Simultaneous: lonazolac (F ex 282 em 345)

REFERENCE
Baeyens,W.R.G.; Van Der Weken,G.; Lievens,L.; Van Overbeke,A. LC-study of lonazolac, naproxen and related non-steroidal anti-inflammatory drugs in a classical and a narrow-bore set-up applying UV and fluorescence detection, *Biomed.Chromatogr.*, **1995**, *9*, 263–264.

SAMPLE
Matrix: solutions

HPLC VARIABLES
Column: 250 × 4.5 5 μm Ultrasphere ODS

Mobile phase: MeCN:10 mM tetrabutylammonium buffer 45:55
Flow rate: 1
Detector: UV 220

CHROMATOGRAM
Retention time: 18.5
Internal standard: ketoprofen (11.6)
Limit of quantitation: 30 ng/mL

REFERENCE
Bischer,A.; Iwaki,M.; Zia-Amirhosseini,P.; Benet,L.Z. Stereoselective reversible binding properties of the glucuronide conjugates of fenoprofen enantiomers to human serum albumin, *Drug Metab.Dispos.*, **1995**, *23*, 900–903.

SAMPLE
Matrix: solutions

HPLC VARIABLES
Column: 250 × 4.6 5 μm Supelcosil LC-DP (A) or 250 × 4 5 μm LiChrospher 100 RP-8 (B)
Mobile phase: MeCN:0.025% phosphoric acid:buffer 25:10:5 (A) or 60:25:15 (B) (Buffer was 9 mL concentrated phosphoric acid and 10 mL triethylamine in 900 mL water, adjust pH to 3.4 with dilute phosphoric acid, make up to 1 L.)
Flow rate: 0.6
Injection volume: 25
Detector: UV 229

CHROMATOGRAM
Retention time: 8.00 (A), 8.87 (B)

OTHER SUBSTANCES
Also analyzed: acebutolol, acepromazine, acetaminophen, acetazolamide, acetophenazine, albuterol, alprazolam, amitriptyline, amobarbital, amoxapine, antipyrine, atenolol, atropine, azatadine, baclofen, benzocaine, bromocriptine, brompheniramine, brotizolam, bupivacaine, buspirone, butabarbital, butalbital, caffeine, carbamazepine, cetirizine, chlorcyclizine, chlordiazepoxide, chlormezanone, chloroquine, chlorpheniramine, chlorpromazine, chlorpropamide, chlorprothixene, chlorthalidone, chlorzoxazone, cimetidine, cisapride, clomipramine, clonazepam, clonidine, clozapine, cocaine, codeine, colchicine, cyclizine, cyclobenzaprine, dantrolene, desipramine, diazepam, diclofenac, diflunisal, diltiazem, diphenhydramine, diphenidol, diphenoxylate, dipyridamole, disopyramide, dobutamine, doxapram, doxepin, droperidol, encainide, ethidium bromide, ethopropazine, fentanyl, flavoxate, fluoxetine, fluphenazine, flurazepam, flurbiprofen, fluvoxamine, furosemide, glutethimide, glyburide, guaifenesin, haloperidol, homatropine, hydralazine, hydrochlorothiazide, hydrocodone, hydromorphone, hydroxychloroquine, hydroxyzine, ibuprofen, imipramine, indomethacin, ketoconazole, ketoprofen, ketorolac, labetalol, levorphanol, lidocaine, loratadine, lorazepam, lovastatin, loxapine, mazindol, mefenamic acid, meperidine, mephenytoin, mepivacaine, mesoridazine, metaproterenol, methadone, methdilazine, methocarbamol, methotrexate, methotrimeprazine, methoxamine, methyldopa, methylphenidate, metoclopramide, metolazone, metoprolol, metronidazole, midazolam, moclobemide, morphine, nadolol, nalbuphine, naloxone, naphazoline, naproxen, nifedipine, nizatidine, norepinephrine, nortriptyline, oxazepam, oxycodone, oxymetazoline, paroxetine, pemoline, pentazocine, pentobarbital, pentoxifylline, perphenazine, pheniramine, phenobarbital, phenol, phenolphthalein, phentolamine, phenylbutazone, phenyltoloxamine, phenytoin, pimozide, pindolol, piroxicam, pramoxine, prazepam, prazosin, probenecid, procainamide, procaine, prochlorperazine, procyclidine, promazine, promethazine, propafenone, propantheline, propiomazine, propofol, propranolol, protriptyline, quazepam, quinidine, quinine, racemethorphan, ranitidine, remoxipride, risperidone, salicylic acid, scopolamine, secobarbital, sertraline, sotalol, spironolactone, sulfinpyrazone, sulindac, temazepam, terbutaline, terfenadine, tetracaine, theophylline, thiethylperazine, thiopental, thioridazine, thiothixene, timolol, tocainide, tolbutamide, tolmetin, trazodone, triamterene, triazolam, trifluoperazine, triflupromazine, trimeprazine, trimethoprim, trimipramine, verapamil, warfarin, xylometazoline, yohimbine, zopiclone

KEY WORDS
also details of plasma extraction

REFERENCE
Koves,E.M. Use of high-performance liquid chromatography-diode array detection in forensic toxicology, *J.Chromatogr.A*, **1995**, *692*, 103–119.

SAMPLE
Matrix: solutions
Sample preparation: 1 mL 5 mM fenoprofen in dichloromethane + 300 µL 1 mg/mL hydroxybenzotriazole in dichloromethane:pyridine 99:1 + 300 µL 11 mg/mL 1-ethyl-3-dimethylaminopropylcarbodiimide in dichloromethane + 300 µL 3.47 mg/mL 1-naphthylamine (Caution! 1-Naphthylamine in a carcinogen!) in dichloromethane, vortex, let stand for 1 h, evaporate to dryness under a stream of nitrogen, reconstitute with 5 mL MeOH, inject an aliquot.

HPLC VARIABLES
Column: 150 × 2.1 Tolycellulose EXP B101 (tris(4-methylbenzoate)cellulose covalently bonded to 10 µm aminopropylsilica)
Mobile phase: MeOH:buffer 85:15 (Buffer was 14.05 g/L sodium perchlorate adjusted to pH 2.0.)
Flow rate: 0.21
Injection volume: 1
Detector: UV 230, UV 254

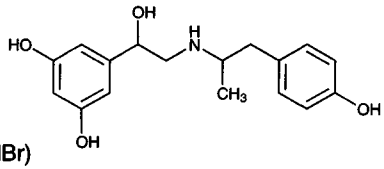
CHROMATOGRAM
Retention time: k' 3.21 (first enantiomer)

OTHER SUBSTANCES
Also analyzed: flurbiprofen, ibuprofen, ketoprofen, tiaprofenic acid

KEY WORDS
derivatization; narrow-bore; chiral; α=1.31; (see Biomed. Chromatogr. 1995; 9; 292)

REFERENCE
Van Overbeke,A.; Baeyens,W.; Van Der Weken,G.; Van de Voorde,I.; Dewaele,C. Comparative chromatographic study on the chiral separation of the 1-naphthylamine derivative of ketoprofen on cellulose-based columns of different sizes, *Biomed.Chromatogr.*, **1995**, *9*, 289–290.

Fenoterol



Molecular formula: $C_{17}H_{21}NO_4$
Molecular weight: 303.36
CAS Registry No.: 13392-18-2, 1944-12-3 (HBr), 1944-10-1 (HBr)
Merck Index: 4022
Lednicer No.: 2 38

SAMPLE
Matrix: solutions
Sample preparation: Prepare a 10 µg/mL solution in MeOH, inject a 20 µL aliquot.

HPLC VARIABLES
Column: 125 × 4.9 Spherisorb S5W silica
Mobile phase: MeOH containing 10 mM ammonium perchlorate and 1 mL/L 100 mM NaOH in MeOH, pH 6.7
Flow rate: 2
Injection volume: 20
Detector: E, LeCarbone, V25 glassy carbon electrode, + 1.2 V

CHROMATOGRAM
Retention time: 1.6

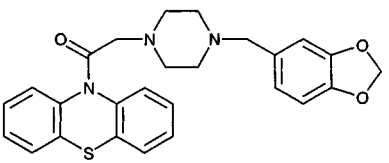
OTHER SUBSTANCES

Also analyzed: acebutolol, acepromazine, acetophenazine, N-acetylprocainamide, albuterol, alprenolol, amethocaine, amiodarone, amitriptyline, antazoline, atenolol, azacyclonal, bamethan, benactyzine, benperidol, benzethidine, benzocaine, benzoctamine, benzphetamine, benzquinamide, bromhexine, bromodiphenhydramine, bromperidol, brompheniramine, brompromazine, buclizine, bufotenine, bupivacaine, buprenorphine, butacaine, butethamate, chlorcyclizine, chlorpheniramine, chlorphenoxamine, chlorprenaline, chlorpromazine, chlorprothixene, cimetidine, cinchonidine, cinnarizine, clemastine, clomipramine, clonidine, cocaine, cyclazocine, cyclizine, cyclopentamine, cyproheptadine, deserpidine, desipramine, dextromoramide, dextropropoxyphene, dicyclomine, diethylcarbamazine, diethylpropion, diethylthiambutene, dihydroergotamine, dimethindene, dimethothiazine, diphenhydramine, diphenoxylate, dipipanone, diprenorphine, dipyridamole, disopyramide, dothiepin, doxapram, doxepin, doxylamine, droperidol, ephedrine, ergocornine, ergocristine, ergocristinine, ergocryptine, ergometrine, ergosine, ergosinine, ergotamine, ethopropazine, etorphine, etoxeridine, fenethazine, fenfluramine, fentanyl, flavoxate, fluopromazine, flupenthixol, fluphenazine, flurazepam, haloperidol, hydroxyzine, hyoscine, ibogaine, imipramine, indapamine, iprindole, isothipendyl, isoxsuprine, ketanserin, laudanosine, lidocaine, lofepramine, loxapine, maprotiline, mecamylamine, meclophenoxate, meclozine, medazepam, mephentermine, mepivacaine, meptazinol, mepyramine, mesoridazine, metaraminol, methadone, methamphetamine, methapyrilene, methdilazene, methotrimeprazine, methoxamine, methoxyphenamine, methoxypromazine, methylephedrine, methylergonovine, methysergide, metoclopramide, metopimazine, metoprolol, mianserin, morazone, nadolol, nalorphine, naloxone, naphazoline, nicotine, nifedipine, nomifensine, nortriptyline, noscapine, orphenadrine, oxeladin, oxprenolol, oxymetazolin, papaverine, pargyline, pecazine, penbutolol, pentazocine, penthienate, pericyazine, perphenazine, phenadoxone, phenampromide, phenazocine, phenbutrazate, phendimetrazine, phenelzine, phenglutarimide, phenindamine, pheniramine, phenmetrazine, phenomorphan, phenoperidine, phenothiazine, phenoxybenzamine, phentolamine, phenylephrine, phenyltoloxamine, physostigmine, piminodine, pimozide, pindolol, pipamazine, pipazethate, piperacetazine, piperidolate, pipradol, pirenzepine, piritramide, pizotifen, practolol, pramoxine, prazosin, prenylamine, prilocaine, primaquine, proadifen, procainamide, procaine, prochlorperazine, procyclidine, proheptazine, prolintane, promazine, promethazine, pronethalol, properidine, propiomazine, propranolol, prothipendyl, protriptyline, proxymetacaine, pseudoephedrine, pyrimethamine, quinidine, quinine, ranitidine, rescinnamine, sotalol, tacrine, terazosin, terbutaline, terfenadine, thenyldiamine, theophylline, thiethylperazine, thiopropazate, thioproperazine, thioridazine, thiothixene, thonzylamine, timolol, tocainide, tolpropamine, tolycaine, tranylcypromine, trazodone, trifluoperazine, trifluperidol, trimeperidine, trimeprazine, trimethobenzamide, trimethoprim, trimipramine, tripelennamine, triprolidine, tryptamine, verapamil, xylometazoline

REFERENCE

Jane,I.; McKinnon,A.; Flanagan,R.J. High-performance liquid chromatographic analysis of basic drugs on silica columns using non-aqueous ionic eluents. II. Application of UV, fluorescence and electrochemical oxidation detection, *J.Chromatogr.*, **1985**, *323*, 191–225.

Fenoverine



Molecular formula: $C_{26}H_{25}N_3O_3S$
Molecular weight: 459.57
CAS Registry No.: 37561-27-6
Merck Index: 4023

SAMPLE

Matrix: blood, urine

Sample preparation: Add 1 mL whole blood or urine to Toxi-Tube A (Toxi-Lab, Irvine CA), add 3 mL water, mix by gentle inversion for 5 min, centrifuge at 1500 g for 5 min. Remove the organic layer and evaporate it to dryness under a stream of nitrogen at 40°, reconstitute the residue with 50 µL MeCN:water 50:50, vortex for 10 s, centrifuge at 7500 g for 2 min, inject a 10 (urine) or 30 (blood) µL aliquot. (The detector wavelength shown is the wavelength of maximum absorbance. This will not necessarily be the optimal wavelength for the separation. Multiple wavelengths from 200-350 nm can be scanned using a diode-array detector. Otherwise, 220 nm may be a reasonable choice for initial work. Matrix may interfere.)

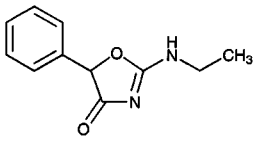
HPLC VARIABLES
Guard column: 20 mm long Symmetry C18
Column: 250 × 4.6 5 μm Symmetry C8 (Waters)
Mobile phase: Gradient. A was 50 mM pH 3.8 sodium phosphate buffer. B was MeCN. A:B 85: 15 for 6.5 min, 65:35 for 18.5 min, 20:80 for 3 min (step gradient), re-equilibrate at initial conditions for 7 min.
Column temperature: 30
Flow rate: 1 for 6.5 min, to 1.5 over 18.5 min, maintain at 1.5 for 3 min (re-equilibrate at 1.5 mL/min)
Injection volume: 10-30
Detector: UV 200.5

CHROMATOGRAM
Retention time: 15.57

KEY WORDS
whole blood

REFERENCE
Gaillard,Y.; Pépin,G. Use of high-performance liquid chromatography with photodiode-array UV detection for the creation of a 600-compound library. Application to forensic toxicology, *J.Chromatogr.A*, **1997**, *763*, 149–163.

Fenozolone



Molecular formula: $C_{11}H_{12}N_2O_2$
Molecular weight: 204.23
CAS Registry No.: 15302-16-6
Merck Index: 4028

SAMPLE
Matrix: blood, urine
Sample preparation: Add 1 mL whole blood or urine to Toxi-Tube A (Toxi-Lab, Irvine CA), add 3 mL water, mix by gentle inversion for 5 min, centrifuge at 1500 g for 5 min. Remove the organic layer and evaporate it to dryness under a stream of nitrogen at 40°, reconstitute the residue with 50 μL MeCN:water 50:50, vortex for 10 s, centrifuge at 7500 g for 2 min, inject a 10 (urine) or 30 (blood) μL aliquot. (The detector wavelength shown is the wavelength of maximum absorbance. This will not necessarily be the optimal wavelength for the separation. Multiple wavelengths from 200-350 nm can be scanned using a diode-array detector. Otherwise, 220 nm may be a reasonable choice for initial work. Matrix may interfere.)

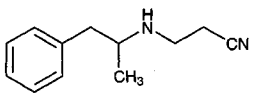
HPLC VARIABLES
Guard column: 20 mm long Symmetry C18
Column: 250 × 4.6 5 μm Symmetry C8 (Waters)
Mobile phase: Gradient. A was 50 mM pH 3.8 sodium phosphate buffer. B was MeCN. A:B 85: 15 for 6.5 min, 65:35 for 18.5 min, 20:80 for 3 min (step gradient), re-equilibrate at initial conditions for 7 min.
Column temperature: 30
Flow rate: 1 for 6.5 min, to 1.5 over 18.5 min, maintain at 1.5 for 3 min (re-equilibrate at 1.5 mL/min)
Injection volume: 10-30
Detector: UV 220.5

CHROMATOGRAM
Retention time: 12.913

KEY WORDS
whole blood

REFERENCE
Gaillard,Y.; Pépin,G. Use of high-performance liquid chromatography with photodiode-array UV detection for the creation of a 600-compound library. Application to forensic toxicology, *J.Chromatogr.A*, **1997**, *763*, 149–163.

Fenproporex



Molecular formula: $C_{12}H_{16}N_2$
Molecular weight: 188.27
CAS Registry No.: 15686-61-0, 18305-29-8 (HCl)
Merck Index: 4036

SAMPLE
Matrix: blood, urine
Sample preparation: Add 1 mL whole blood or urine to Toxi-Tube A (Toxi-Lab, Irvine CA), add 3 mL water, mix by gentle inversion for 5 min, centrifuge at 1500 g for 5 min. Remove the organic layer and evaporate it to dryness under a stream of nitrogen at 40°, reconstitute the residue with 50 µL MeCN:water 50:50, vortex for 10 s, centrifuge at 7500 g for 2 min, inject a 10 (urine) or 30 (blood) µL aliquot. (The detector wavelength shown is the wavelength of maximum absorbance. This will not necessarily be the optimal wavelength for the separation. Multiple wavelengths from 200-350 nm can be scanned using a diode-array detector. Otherwise, 220 nm may be a reasonable choice for initial work. Matrix may interfere.)

HPLC VARIABLES
Guard column: 20 mm long Symmetry C18
Column: 250 × 4.6 5 µm Symmetry C8 (Waters)
Mobile phase: Gradient. A was 50 mM pH 3.8 sodium phosphate buffer. B was MeCN. A:B 85:15 for 6.5 min, 65:35 for 18.5 min, 20:80 for 3 min (step gradient), re-equilibrate at initial conditions for 7 min.
Column temperature: 30
Flow rate: 1 for 6.5 min, to 1.5 over 18.5 min, maintain at 1.5 for 3 min (re-equilibrate at 1.5 mL/min)
Injection volume: 10-30
Detector: UV 200.5

CHROMATOGRAM
Retention time: 19.263

KEY WORDS
whole blood

REFERENCE
Gaillard,Y.; Pépin,G. Use of high-performance liquid chromatography with photodiode-array UV detection for the creation of a 600-compound library. Application to forensic toxicology, *J.Chromatogr.A*, **1997**, *763*, 149–163.

SAMPLE
Matrix: solutions

HPLC VARIABLES
Column: 250 × 4.6 Zorbax RX
Mobile phase: Gradient. A was 10 mL concentrated orthophosphoric acid and 7 mL triethylamine in 1 L water. B was 10 mL concentrated orthophosphoric acid and 7 mL triethylamine in 200 mL water, make up to 1 L with MeCN. A:B from 100:0 to 0:100 over 30 min, maintain at 0:100 for 5 min.
Column temperature: 30
Flow rate: 2
Detector: UV 210

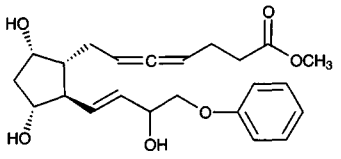
OTHER SUBSTANCES

Also analyzed: acepromazine, acetaminophen, acetophenazine, albuterol, aminophylline, amitriptyline, amobarbital, amoxapine, amphetamine, amylocaine, antipyrine, aprobarbital, aspirin, atenolol, atropine, avermectin, barbital, benzocaine, benzoic acid, benzotropine, benzphetamine, berberine, bibucaine, bromazepan, brompheniramine, buprenorphine, buspirone, butabarbital, butacaine, butethal, caffeine, carbamazepine, carbromal, chloramphenicol, chlordiazepoxide, chloroquine, chlorothiazide, chloroxylenol, chlorphenesin, chlorpheniramine, chlorpromazine, chlorpropamide, chlortetracycline, cimetidine, cinchonidine, cinchonine, clenbuterol, clonazepam, clonixin, clorazepate, cocaine, codeine, colchicine, cortisone, coumarin, cyclazocine, cyclobenzaprine, cyclothiazide, cyheptamide, cymarin, danazol, danthron, dapsone, debrisoquine, desipramine, dexamethasone, dextromethorphan, dextropropoxyphene, diamorphine, diazepam, diclofenac, diethylpropion, diethylstilbestrol, diflunisal, digitoxin, digoxin, diltiazem, diphenhydramine, diphenoxylate, diprenorphine, dipyrone, disulfiram, dopamine, doxapram, doxepin, dronabinol, ephedrine, epinephrine, epinine, estradiol, estriol, estrone, ethacrynic acid, ethosuximide, etonitazene, etorphine, eugenol, famotidine, fenbendazole, fencamfamine, fentanyl, flubendazole, flufenamic acid, flunitrazepam, 5-fluorouracil, fluoxymesterone, fluphenazine, furosemide, gentisic acid, gitoxigenin, glipizide, glunixin, glutethimide, glybenclamide, guaiacol, halazepam, haloperidol, hydrochlorothiazide, hydrocodone, hydrocortisone, hydromorphone, hydroxyquinoline, ibogaine, ibuprofen, iminostilbene, imipramine, indomethacin, isocarbostyril, isocarboxazid, isoniazid, isoproterenol, isoxsuprine, ivermectin, ketamine, ketoprofen, kynurenic acid, levorphanol, lidocaine, lorazepam, lormetazepam, loxapine, mazindol, mebendazole, meclizine, meclofenamic acid, medazepam, mefenamic acid, megestrol, mepacrine, meperidine, mephentermine, mephenytoin, mephesin, mephobarbital, mepivacaine, mescaline, mesoridazine, methadone, methamphetamine, methapyrilene, methaqualone, methazolamide, methocarbamol, methoxamine, methsuximide, methyl salicylate, methyldopa, methyldopamine, methylphenidate, methylprednisolone, methyltestosterone, methyprylon, metoprolol, mibolerone, morphine, nadolol, nalorphine, naloxone, naltrexone, naphazoline, naproxen, nefopam, niacinamide, nicotine, niacin, nifedipine, niflumic acid, nitrazepam, norepinephrine, nortriptyline, noscapine, nylidrin, oxazepam, oxycodone, oxymorphone, oxyphenbutazone, oxytetracycline, papaverine, pargyline, pemoline, pentazocine, pentobarbital, persantine, phenacetin, phenazocine, phenazopyridine, phencyclidine, phendimetrazine, phenelzine, pheniramine, phenobarbital, phenothiazine, phensuximide, phentermine, phenylbutazone, phenylephrine, phenylpropanolamine, piperocaine, prazepam, prednisolone, primidone, probenecid, progesterone, propiomazine, propranolol, propylparaben, pseudoephedrine, puromycin, pyrilamine, pyrithyldione, quazepam, quinaldic acid, quinidine, quinine, ranitidine, recinnamine, reserpine, resorcinol, saccharin, albuterol, salicylamide, salicylic acid, scopolamine, scopoletin, secobarbital, strychnine, sulfacetamide, sufadiazine, sulfadimethoxine, sulfaethidole, sulfamerazine, sulfamethazine, sulfamethoxizole, sulfanilamide, sulfapyridine, sulfasoxizole, sulindac, tamoxifen, temazepam, testosterone, tetracaine, tetracycline, tetramisole, thebaine, theobromine, theophylline, thiabendazole, thiamine, thiamylal, thiobarbituric acid, thioridazine, thiosalicylic acid, thiothixene, thymol, tolazamide, tolazoline, tobutamide, tolmetin, tranylcypromine, triamcinolone, tribenzylamine, trichloromethiazide, trifluoperazine, trihexyphenidyl, trimethoprim, tripelennamine, triprolidine, tropacocaine, tyramine, verapamil, vincamine, warfarin, yohimbine, zoxazolamine

REFERENCE

Hill,D.W.; Kind,A.J. Reversed-phase solvent gradient HPLC retention indexes of drugs, *J.Anal.Toxicol.*, **1994**, *18*, 233–242.

Fenprostalene



Molecular formula: $C_{23}H_{30}O_6$
Molecular weight: 402.49
CAS Registry No.: 69381-94-8
Merck Index: 4037
Lednicer No.: 4 9

SAMPLE

Matrix: solutions

HPLC VARIABLES
Column: 250 × 3.2 10 μm Spherisorb ODS
Mobile phase: MeOH:20 mM acetic acid 55:45
Injection volume: 100
Detector: UV 219 or 270

CHROMATOGRAM
Retention time: 7

OTHER SUBSTANCES
Simultaneous: degradation products

REFERENCE
Johnson,D.M.; Taylor,W.F.; Thompson,G.F.; Pritchard,R.A. Degradation of fenprostalene in aqueous solution, *J.Pharm.Sci.*, **1983**, *72*, 946–948.

SAMPLE
Matrix: solutions

HPLC VARIABLES
Column: 250 × 4.6 5 μm Ultrasphere octyl
Mobile phase: MeCN:water 35:65 to 45:55
Injection volume: 100
Detector: UV 219

CHROMATOGRAM
Retention time: 22

REFERENCE
Johnson,D.M.; Taylor,W.F. Degradation of fenprostalene in polyethylene glycol 400 solution, *J.Pharm.Sci.*, **1984**, *73*, 1414–1417.

SAMPLE
Matrix: tissue
Sample preparation: Homogenize (Polytron Model PT-10-ST/PT-35) 60 g liver with 240 mL cold water, adjust to pH 2 with 2 M HCl, extract three times with an equal volume of ethyl acetate. Combine the extracts, neutralize with solid sodium bicarbonate, dry over anhydrous sodium sulfate, evaporate to 50 mL under vacuum, evaporate to dryness, take up the residue in 50 mL MeOH:water 90:10, wash with 40 mL hexane, evaporate the MeOH/water phase to dryness, dissolve the residue in MeOH, chromatograph on silica gel 60F-254 tlc plates (EM Science) with ethyl acetate:MeOH 80:20, scrape off the fenprostalene layer, desorb with 100 μL mobile phase, inject a 70 μL aliquot.

HPLC VARIABLES
Column: μBondapak C18
Mobile phase: MeOH:10 mM acetic acid 60:40
Flow rate: 1
Injection volume: 70
Detector: UV 225

KEY WORDS
pig; liver

REFERENCE
Spires,H.R.; Bowen,J.L.; Tomlinson,R.V.; Donahue,D.J. Pharmacokinetic and tissue residue characteristics of fenprostalene, a prostaglandin F2 α analog, in swine, *Am.J.Vet.Res.*, **1990**, *51*, 386–390.